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Analysis of extrusion foaming of polyolefins using isobutane and CO₂

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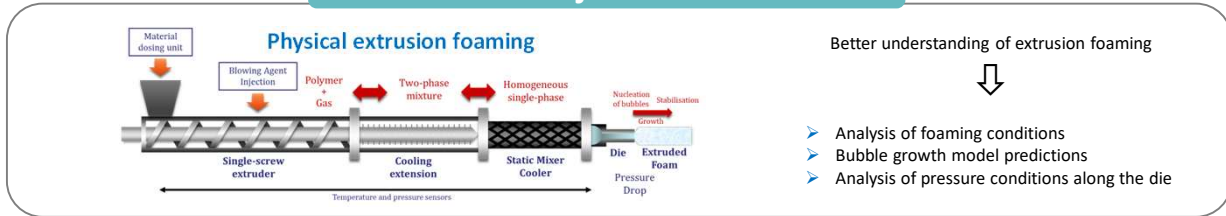


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Objective



Materials, foaming conditions

Two sets of foaming experiments:

LDPE grades:

CD0230 and LD0304 from TotalEnergies
Foaming with 5 and 8 wt% iso-butane

Range of T_{melt} during foaming: 120 to 100 °C at the die exit

CD0230 and LD0304: $T_c = 97^\circ\text{C}$ (start 100°C)

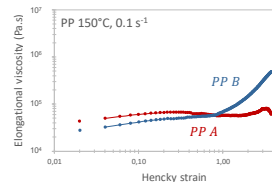
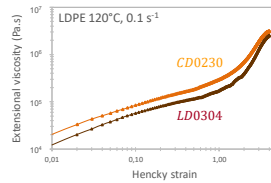
PP experimental grades:

PP A (linear chains) and PP B (branched chains) from TotalEnergies
Foaming with 1 and 2 wt% CO₂

Range of T_{melt} : 170 to 130°C at the die exit

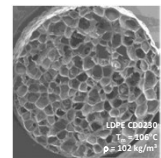
PP A: $T_c = 97^\circ\text{C}$ (start 105°C), PP B: $T_c = 104^\circ\text{C}$ (start 110°C)

Rheological behaviour



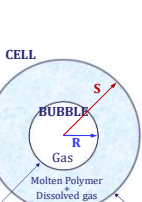
Foam characterization:

- SEM → Cell density (cells/m³)
- Mean bubble size
- Foam density (kg/m³)



Bubble growth modeling

Model equations and hypotheses [1]



Growth of a gas bubble in a polymer shell containing polymer and dissolved gas

Constant cell density: $n_{b,0} \rightarrow S_0 = \sqrt[3]{\frac{3}{4\pi n_{b,0}}}$ $R_0 = 1\mu\text{m}$ $c(r,0) = c_0$

Diffusion equation $\frac{\partial c}{\partial t} + \frac{R}{r^2} \frac{\partial c}{\partial r} = \frac{D}{r^2} \frac{\partial}{\partial r} \left(r^2 \frac{\partial c}{\partial r} \right)$

Bubble growth $\frac{d}{dt} (p_g R^3) = 3 D R T \frac{\partial c}{\partial r} \bigg|_{r=R}$

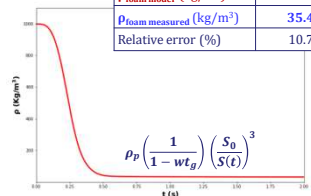
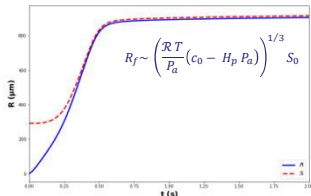
Momentum equation $P_g - P_a = \frac{2\gamma}{R} - \tau_{rr}(S) - \frac{2}{3} \int_0^S \dot{\gamma}^2 - \dot{\gamma}_0^2 \frac{\tau_{rr} - \tau_{\theta\theta}}{r^3} dy$

Viscoelastic matrix: Multi-Maxwell or Phan-Thien-Tanner model

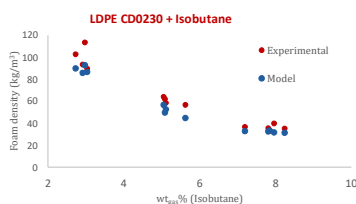
Bubble growth prediction

CD0230	%wt isobutane	8.23
	T_{melt} (°C)	104
	$n_{b,0}$ (cell/cm ³)	9500
	D (m ² /s)	1.23E-09
	H_p (mol/(m ³ Pa))	1.818E-03

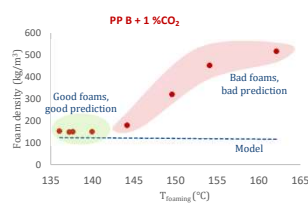
$\rho_{ppolymer}$ (kg/m ³)	922
$\rho_{foam model}$ (kg/m ³)	31.6
$\rho_{foam measured}$ (kg/m ³)	35.4
Relative error (%)	10.7



Foam density → comparison between model predictions and experiments

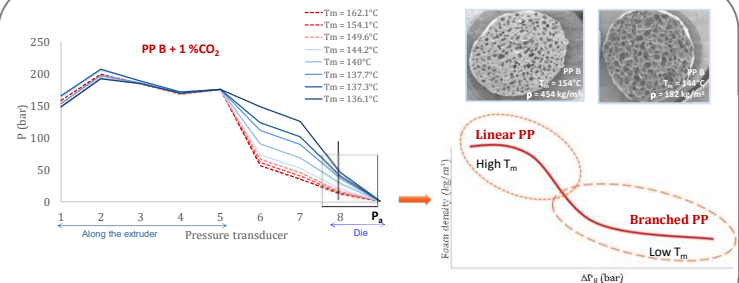


Low density foams → Well predicted by the bubble growth model



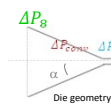
Different densities of PP foams: → Analysis of pressure drop in the die

Analysis of pressure drop in the die



Why analysing ΔP in the die ?

Pressure drop in the die = responsible for the foam formation (nucleation + growth)



$\Delta P_{cis} = \Delta P_{conv} + \Delta P_{cap}$ $\Delta P_{conv} = \frac{2KL_c}{3m(R_0 - R_1)} \left(\frac{3m+1}{m} \frac{Q}{\pi} \right)^m \left[\frac{1}{R_1^{3m}} - \frac{1}{R_0^{3m}} \right]$ [2]

$\Delta P_{cap} = 2KL_f \left(\frac{3m+1}{m} \frac{Q}{\pi} \right)^m \frac{1}{R_1^{(1+3m)}}$ Power law: K, m

If $\Delta P_8 > \Delta P_{cis} \Rightarrow$ Additional contribution: $\Delta P_{el} = \Delta P_8 - \Delta P_{cis}$ (Eq. 1)

Steps :

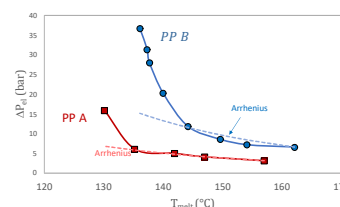
- Calculation of ΔP_{cis} ✓
- Comparison with ΔP_8 ✓
If $\Delta P_8 > \Delta P_{cis} \Rightarrow$ calculation $\Delta P_{el} = \Delta P_8 - \Delta P_{cis}$ (Eq. 1)
- If viscoelastic effect, $\Rightarrow \Delta P_{el}$ variation with T should follow an Arrhenius law [3]
- If $\Delta P_{el}(T) >$ Arrhenius law \Rightarrow Crystallization at the origin of ΔP_{el} excess
 \Rightarrow Calculation of η_{el} (Eq. 2)
 \Rightarrow Estimation of a crystallized fraction ϕ (Eq. 3)

$\eta_{el} = \frac{\pi \Delta P_{el} (1 - \cos \alpha)}{Q \sin^3 \alpha} \frac{R_0^2 R_1^2}{(R_1 + R_0) \left(\ln \frac{R_1}{R_0} \right)}$ (Eq. 2)

[3]

$\eta_{susp} = \eta_{matrix} \left[1 - \frac{\phi}{B} \right]^{-2}$ (Eq. 3)

$B = 0.54$ for spherulites [4]



- Existence of an additional contribution (ΔP_{el}) in the die
- Low $\Delta P_{el} = f(T)$ (higher T) follow an Arrhenius dependence
- The presence of a crystalline state in the die could explain the pressure drop excess and help in some cases the foaming process

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- Agassant J.F. et al., "Mise en forme des polymères-approche thermomécanique de la plasturgie" Lavoisier, (2015)
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